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METHOD FOR THE MANUFACTURE OF GRANULAR OR POWDERED XYLITE

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### Detailed explanation of the invention

The present invention relates to a method for the manufacture of granular or powdered xylite. Xylite is a type of sugar alcohol obtained by the high-pressure reduction of D-xylose and does not exist in nature. The molecular formula is  $C_5H_{10}O_5$  and it has a melting point of  $94.5^{\circ}C$ . Xylite is chemically stable. It is cool to the touch and the same sweetness as sucrose. It has a low hygroscopicity and is readily soluble in water. Furthermore, the browning phenomenon (Maillard reaction) of amino acids does not occur. Xylite is commonly used as a sugar source for diabetics or liver-disease patients in the form of injection solutions or tablets or as transfusion solutions before or after operations.

High-purity xylite crystals for injection and transfusion-solution uses mentioned previously can be obtained by the addition of seed crystals and a solvent into [as] a 75-80% aqueous solution of xylite, then slowly allowing it to cool. On the other hand, xylite crystals with a relatively low purity for tablets mentioned previously can be manufactured by the following conventional method. In other words, the aqueous xylite solution is concentrated to a water content of less than 1%. In a sense, this xylite melt is stirred as such as at specified temperature. Alternatively, to the molten xylite mentioned previously, a xylite powder is added at 3-30% as seed crystals. Then it is stirred at a specified temperature in the same manner. The microcrystals are sufficiently deposited and grown. This is then allowed to cool. After it has been completely converted into a

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solid of microcrystals, this solid is pulverized or crushed to form granular or powdered crystalline xylite.

In other words, this conventional method for the manufacture of granular or powdered xylite is, in essence, a method in which, during the deposition growth of microcrystals from the xylite melt or after the completion of deposition growth, this is allowed to cool to cause complete solidification; it is then pulverized or crushed to form granules or powders. However, this conventional method has a major drawback because a long period of time, more than 10 h, is required in the cooling solidification process mentioned previously. Furthermore, for this solid, deposition growth of crystals is not complete and hygroscopicity and caking characteristics occur readily.

As a result of investigations to solve the drawbacks of the conventional method described previously, the present inventor has discovered that when the material obtained by sufficiently carrying out the deposition growth of microcrystals, with or without the addition of seeds into the melt of xylite or its concentrated aqueous solution in the same manner as the conventional method to start with, is maintained under an appropriate temperature and stirring state in continuation, the microcrystals maintain plasticity and the whole substance will be in the form of a soft lump, and this state is maintained. Furthermore, this soft, plasticizable lump can be directly molded into granules using an appropriate apparatus. Thus, when a soft, plasticizable microcrystalline lump of xylite mentioned previously is molded into a granular form, its surface area is remarkably increased. Because of this, the cooling, drying, and crystallization of the plasticizable lump are promoted.

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Crystalline xylite solidified to a granular form can be obtained in a short period of time. Furthermore, by further crushing, powdered xylite can be obtained.

The method of the present invention will be further explained in detail. In other words, a concentrated material or a melt obtained by the concentration of an aqueous xylite solution to remove water to less than about 15%, in a high-temperature state, is allowed to flow continuously into an appropriate continuous-stirring apparatus, such as a kneader, for mixing at a specified temperature to cause the deposition and growth of microcrystals. Alternatively, to the introduced xylite concentrated solution or the melt described previously, powdered xylite crystals at about 1-50% of the weight of the solid are added as seeds. At the same specified temperature, mixing is conducted to promote the deposition and growth of crystals. The temperature of the concentrated solution or the melt of xylite mentioned previously is from about 60°C to 94.5°C, the melting point of xylite. If it is less than 60°C, mixing cannot be conducted smoothly since the viscosity is too high even at the upper limit, 15%, of the water content. Moreover, if it is above the melting point, crystallization does not occur even if the water content is 0%. If the amount of addition of the seeds mentioned previously is more than 50% of the solid in the xylite concentrated solution or the melt, the soft plasticity of the mixture as a whole will be lost even at 15%, the maximum limit of the water content in xylite mentioned previously. It will be impossible to carry out molding into a granular shape or a vermicelli shape. With no addition of the seeds mentioned previously, the deposition and growth of microcrystals of xylite

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will be somewhat slowed down in comparison to when seeds are added.

In regard to properties of the mixture allowed to flow into the kneader mentioned previously, there is a directly proportional relationship between the water content and temperature of the xylite concentrated material or melt with the amount of the seeds added into this. In other words, with an increase in the water content or an increase in temperature, it is necessary to add a larger amount of the seeds. Similarly, there is a correlation between the water content and temperature previously mentioned. If the water content is low, it is impossible to lower the temperature to a large extent. For example, in the case of less than 3% water content, a temperature of more than about 80°C is required. The location of addition of the seeds into the kneader may be upstream or downstream of the flow inlet of the xylite solution or melt. The amount of addition of the seeds advantageous to the industry is about 10%.

The xylite concentrated material or melt allowed to flow in this manner is a liquid at the beginning. Afterwards, by mixing and stirring at a specified temperature, fine crystals are gradually deposited and grown. A soft lump with plasticity as a whole is formed. Also, if this soft, plasticizable microcrystalline lump is maintained at an appropriate temperature and in a stirred state, the state of the plasticizable lump mentioned previously can be continued. The optimum time for mixing in the kneader is about 5-20 min.

As described previously, when the whole substance after sufficient progress of the crystallization of xylite becomes a soft crystal-particle lump having plasticity, this is

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continuously sent into an appropriate molding machine, such as a screw-extension molding machine, to be extruded into a vermicelli shape from the injection part of the same machine. Since the surface area of this injection molded material is enlarged a few tens to a few hundreds times that of the plasticizable, microcrystalline particle lump mentioned previously, the vermicelli-shaped plasticizable material is rapidly cooled and dried. At the same time, the growth of crystals is also accelerated. The vermicelli-shaped xylite mentioned previously preferably has a diameter of 1-3 mm and a length of about 200-500 mm. This cooled vermicelli-shaped material is then introduced into an appropriate granule-adjusting machine, such as a flash mill, to achieve cutting and particle adjustment to a constant length. In this case, if the size of the injection hole of the extrusion-type molding machine for obtaining the vermicelli shape mentioned previously and the cut length due to the granule-adjusting machine afterwards are selected to be in agreement with each other, granular crystalline xylite with a constant size and shape as such can be obtained. These granules are crushed and powders can be readily obtained.

In the method of the present invention, to obtain the state of a soft, plasticizable crystal-particle lump by the mixing of the previously mentioned xylite concentrated material or melt, since xylite has difficulty in crystallization compared to glucose or other saccharides, the state of such a plasticizable, microcrystalline-particle lump can be relatively readily obtained. In other words, the present invention utilizes the property of the relative difficulty in crystallization of xylite, and the state of a soft, plasticizable microcrystalline-particle

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lump can be readily obtained. This is then immediately molded as such into a granular form with a large surface area. In doing this, granular or powdered xylite can be very rapidly manufactured.

The granules or powders of xylite manufactured by the method of the present invention have higher melting points than those obtained by the conventional method. For example, with respect to a xylite melt with a water content of 1% seeds at 10% of its solid content are added. This is mixed and stirred at 70-75°C for 10 min. It is then extruded into a vermicelli shape with a molding machine. After cooling to 40°C, it is cut and the melting of the granular product obtained reaches 92-93.5°C. Furthermore, the xylite obtained according to the method of the present invention has a low hygroscopicity and caking characteristics but an excellent fluidity. Packaging and storage are also easy.

Next, the present invention will be further explained in detail with application examples.

#### Application Example 1

An aqueous xylite solution with a purity of about 98% was concentrated at a temperature of 120°C to a water content of less than 1%. The temperature of this xylite melt was lowered to about 90°C. It was allowed to flow into a continuous kneader at a rate of 1 kg/min. Powdered xylite was added as seeds into this at a rate of 0.1 kg/min. It was mixed and stirred at a temperature of 70-80°C. A soft, ripened plasticizable lump with the sufficient growth of microcrystals for a residence time of



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about 12 min was obtained. Next, this was extruded into a vermicelli form with a diameter of 2 mm and a length of 30-50 cm with a screw-extrusion molding machine. After this molded product had been cooled to 40°C, it was cut to a constant length with a flash mill. The material obtained by crushing of the resulting granular xylite was a crystalline powder with a water content of less than 1% and a melting point of 92-93.5°C.

#### Application Example 2

An aqueous xylite solution with a purity of about 98% was concentrated to a water content of about 13%. The temperature of this concentrated solution was lowered to about 80°C. It was allowed to flow into a continuous kneader at a rate of 1 kg/min. Powdered xylite was added as seeds into this at a rate of 0.1 kg/min. It was mixed and stirred at a temperature of 60-70°C. A soft, plasticizable lump obtained by the sufficient growth and ripening of microcrystals for a residence time of about 20 min was obtained. By the same treatment as that in the previous example for this hereafter, it was molded into a vermicelli form. This molded product was dried with hot air over 30 min at a temperature of 70-80°C. After it was cooled to 40°C, it was cut to a constant length with a flash mill. The granular xylite obtained was a crystalline solid with a water content of less than 1% and a melting point of 91-93°C.

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### Application Example 3

An aqueous xylite solution with a purity of about 98% was concentrated to a water content of less than about 1%. The temperature of this xylite melt was lowered to about 80°C. It was allowed to flow into a continuous kneader at a rate of 1 kg/min. It was mixed and stirred at a temperature of about 70°C. A soft, ripened plasticizable lump obtained by the sufficient deposition and growth of microcrystals for a residence time of about 18 min was obtained. The granular xylite obtained by the same treatment as in Application Example 1 for this was a crystalline solid with a water content of less than 1% and a melting point of 91-93°C.

### Claims

1. A method for the manufacture of granular or powdered xylite characterized by the fact that a xylite concentrated aqueous solution with a water content of less than 15% or a melt as such is mixed and stirred at a temperature above 60°C and below the melting point of xylite so that the whole substance is converted into a soft, plasticizable microcrystalline-particle lump. This is then sent into an appropriate molding machine for extrusion into a vermicelli shape; this vermicelli-shaped material is cooled to a temperature below about 60°C, and is then cut to a specified length.

2. A method for the manufacture of granular or powdered xylite characterized by the fact that, in a xylite concentrated aqueous solution with a water content of less than 15% or when a

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melt, powdered or granular xylite at 1-50% of the solid's content of this aqueous solution or melt is added, it is mixed and stirred at a temperature about 60°C and below the melting point of xylite so that the whole substance is a soft, plasticizable microcrystalline-particle lump, with this then being sent into an appropriate molding machine for extrusion into a vermicelli form, and with this vermicelli-shaped material being cooled to a temperature below about 60°C, then cut to a specified length.